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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of: Yasushi Akiyama

Serial No.: 10 / 519,242

Group Art Unit: 1724

Filed: December 22, 2004

Examiner: WU, IVES J

For: Composition for antireflection coating and method for forming pattern

**Commissioner for Patents****P. O. Box 1450****Alexandria, VA 22313-1450**DECLARATION UNDER 37 C. F. R. § 1.132

I, Yasushi AKIYAMA, a Japanese citizen, residing in Kakegawa-shi, Japan, declare as follows:

I received a Bachelor of Science degree from Yokohama City University in March, 1999 and a Master of Science degree in organic chemistry from Yokohama City University in March, 2001. I had been employed by Clariant (Japan) K.K. as a research & development engineer from April, 2001 to October, 2004, and then, I have been employed by AZ Electronic Materials (Japan) K.K. as a research & development engineer since October, 2004. I have been involved in research and development work relating to photoresists and ancillary chemicals since 2001.

I have been named as an inventor on at least 2 US pending Patents and 3 Japanese pending patent applications. I am an applicant in the above-mentioned patent application and an inventor of the invention claimed therein. I am now familiar with the references which were used in the rejection thereof.

The following experiments were performed by me or under my direction and control by a colleague who has communicated his finding to me. The experiments were conducted to find out the effect of using high concentrations of acetic acid in the present

inventive composition.

### EXPERIMENTAL REPORT

Review of using a mixture of low-grade alkylcarboxylic acid such as acetic acid with water in the present inventive composition. The prior art discloses the use of 20 and 30 weight% of acetic acid in a composition.

#### Test 1

##### Example 1

2.56 parts by weight of a fluorine-containing polymer having a number-average molecular weight of 4,700 and a weight-average molecular weight of 6,300 as determined using polystyrene standards and represented by the following general formula (I):



was dispersed in 95.21 parts by weight of water at an ordinary temperature to prepare a slurry, then 0.23 part by weight of monoethanolamine was gradually added to prepare an aqueous antireflective coating composition. Further, 0.17 part by weight of alkylsulfonicacid which is mixture of C14 – C18 alkylsulfonicacid and 1.83 parts by weight of acetic acid was added thereto to prepare a total weight of 100 parts by weight of a uniform aqueous solution. The solubility of ingredients are shown in the following Table 1(B). The resultant uniform aqueous solution was filtered through a 0.1  $\mu\text{m}$  filter to obtain an anti-reflective coating composition.

Separately, a positive-working photoresist, AZ DX5240P ("AZ" being a registered trademark), comprising an acetal type polymer and made by AZ Electronic Materials (Japan) K.K. was coated on a silicon wafer using a spin coater (Mark 8) made by Tokyo Electron Co., Ltd., followed by pre-baking on a hot plate at 90°C for 60 seconds to form a 5850 Å photoresist film on the silicon wafer. The thickness of the film was measured by means of a film thickness-measuring apparatus (SM300) made by Prometric Co.

Subsequently, the above-described anti-reflective coating composition was applied on the photoresist film using the same spin coater, followed by pre-baking on a

hot plate at 90°C for 60 seconds to form a 450 Å thick anti-reflective coating film. After conducting a post exposure bake on a hot plate at 120°C for 60 seconds, paddle development was conducted using an alkaline developer, AZ 300MIF developer ("AZ" being a registered trademark; a 2.38% by weight aqueous solution of tetramethylammonium hydroxide), made by AZ Electronic Materials (Japan) K.K. as a developer at 23°C for 1 minute. The thickness of the developed photoresist was measured using the same film thickness-measuring apparatus as described above.

The value of film thickness of the thus-obtained photoresist after development was subtracted from the value of film thickness of the photoresist before development, and the remainder value was taken as the amount of reduction in thickness of the film. Further, the value of film thickness at the periphery of the thus-obtained photoresist after development was subtracted from the value of film thickness at the center of the photoresist after development, and the remainder value was taken as the difference of resist film-thickness between the center and the periphery.

Also, the refractive index of the anti-reflective film at a wavelength of 248 nm are shown in the following Table 1(B).

#### Examples 2 to 4

The same procedures as in Example 1 except for changing the amount (parts by weight) of ingredients was conducted as shown in the following Table 1 (A) .

Table 1(A) Formulation

	fluorine- containing polymer	alkyl sulfonic acid	acetic acid	monoethanol amine	D.I.Water	total
Example1	2.56	0.17	1.83	0.23	95.21	100.00
Example2	2.56	0.17	0.00	0.23	97.04	100.00
Example3	2.56	0.17	20.00	0.23	77.04	100.00
Example4	2.56	0.17	30.00	0.23	67.04	100.00

#### Results

The results of the tests for Examples 1-4 are shown in the following Table1(B).

Table 1(B) Test Results

DX 5240P					
	refractive index	solubility	Thickness of resist (Å)	Reduction in amount of photoresist film (Å)	Difference of photoresist film-thickness between the center and the periphery (Å)
Example1	1.45	dissolved	5850.6	687.4	48.5
Example2	1.45	dissolved	5817.3	672.8	40.5
Example3	1.45	dissolved	5855.9	908.1	148.5
Example4	1.45	dissolved	5855.7	1381.1	359.7

As shown in the above Table1 (B) the acetic acid concentration of greater than 20 weight %, Example 3 and 4, gave a very large reduction in thickness of the photoresist film as compared to Examples 1 and 2, and a large negative difference of resist film-thickness between the center of resist film and the periphery of resist film. This result shows that in our present invention using a controlled amount of acid unexpectedly gives far superior results than the amounts in the prior art. The prior art suggests using high concentrations of acetic acid, around 20 to 30 weight%, which in the present invention give a large reduction in the photoresist film and also unacceptable nonuniformity of the photoresist film.

I declare that all statements herein made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

October 10, 2007

(date)

Yasushi Akiyama

Yasushi AKIYAMA